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Report 2192

DETERMINATION OF CONTAMINANTS IN LESS-FLAMMABLE HYDRAULIC FLUIDS

September 1976

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U.S. ARMY MOBILITY EQUIPMENT
RESEARCH AND DEVELOPMENT COMMAND
FORT BELVOIR, VIRGINIA

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

The introduction of a new, less-flammable, synthetic-base hydraulic fluid (MIL-H-46170) for armored vehicles operating in hostile environment created a problem in the operability of the M60 A1 (AOS) hydraulic and gun-control system. In the determining of problem origin, a need was created for methodology to (1) determine the level of MIL-H-6083 petroleum-base hydraulic fluid remaining in the system after retro-fit and (2) determine the amount of adsorbed water. Gas-liquid chromatographic procedures were subsequently developed to accomplish these objectives.

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CONTENTS

ILLUSTRATIONS

TABLES

DETERMINATION OF CONTAMINANTS IN LESS-FLAMMABLE HYDRAULIC FLUIDS

I. INTRODUCTION

In March 1974, the Commanding General of the Army Materiel Command (AMC) (now DARCOM) made a command decision to replace the petroleum-base hydraulic fluid, MIL-H-6083 (OHT),[1] specified for Army ground equipment with a recently developed synthetic, hydrocarbon-base hydraulic fluid described by Purchase Description FA PD-5136, Hydraulic Fluid, Rust Inhibited, Fire Resistant, Synthetic Hydrocarbon (FRH). This changeover for Army armored equipment occurred as a result of reports received in December 1973 from the Mid-East Conflict inferring that the M60A1 (AOS) tanks are particularly vulnerable to turret fires due to ignition of the OHT fluid used in the M140 gun mount and turret/gun-control system. In May 1974, AMC directed the Troop Support Command (TROSCOM) to purchase the FA PD-5136 fluid. Subsequently, the Department of the Army issued a message to all commands requesting replacement of OHT with the FRH fluid initially in the M60A1 (AOS) tanks followed by other armored equipment (i.e., M60A2, M48A1, etc).

Early in 1975, a series of field problems occurred involving the FRH fluid. A task force was immediately formed to investigate the problem. The preliminary findings revealed, among other things, a significant level of residual OHT contamination to be present in those tanks retrofitted with FRH fluid. This had considerable significance in view of the initial flammability assessments where 5% OHT contamination decreased the fire-retardant characteristics of new FRH fluids.[2]

The major problem subsequently identified by the task force involved malfunctions of the gunner's handle control assembly located in the hydraulic turret/gun-control system. However, this problem did not exist in all new M60A1 (AOS) tanks utilizing the FRH fluid. The FRH hydraulic fluid task force determined that the power spool valve was the principal offender and that the problem occurred only on new M60A1 (AOS) tanks using one of two products qualified under the FA PD-5136 Purchase Description. Subsequent examination of several spool valves that failed revealed the presence of a sticky residue which was easily removed.

Laboratory experiments with the one suspect FRH fluid used as the "factory-fill" for new tanks led to the hypothesis that trace moisture reacted with a proprietary

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[1] MIL SPEC MIL-H-6083, *Hydraulic Fluid, Petroleum Base, for Preservation and Operation, 9/28/73.*

[2] B. R. Wright, W. D. Weatherford, and M. E. LePera, "Investigation of the Flammability Properties of Hydraulic Fluids," *Final Report AFLRL No. 70,* 6/75.

water solubilizer additive which, in turn, caused the rust inhibitor (barium dinonylnaphthalene sulfonate) to precipitate from solution. It should be noted that the other qualified fluid did not contain this water solubilizer additive and consequently did not "react" with trace moisture.

To support this hypothesis, a humidification test developed for automotive hydraulic brake fluids was used.[3] The general details of this humidification test procedure are provided in the Appendix. However, in the course of investigating the phenomena, a need was generated for accurate measurements of water adsorption. The Karl Fisher (KF) technique (ASTM D1744)[4] was initially explored but found to be unsuitable because of a reaction occurring between the KF reagents and the synthetic, hydrocarbon-base hydraulic fluid.

In view of the two problem areas, it became necessary for test methods to be developed for the quantitative determination of residual OHT in FRH fluid and adsorbed water in FRH fluid. Gas chromatographic techniques were subsequently explored as the means to accomplish the project objectives.[5] [6]

II. DETAILS OF TEST

1. Chromatograph. The equipment used to obtain the chromatographs was a Model 810 Linear Programed Temperature Gas Chromatograph (F&M Scientific Corp) equipped with a Brown Electronik Recorder (Minneapolis-Honeywell).

2. Column Preparations. An 18.288-decimeter (6-ft) length of 6-mm (0.25-in.) diameter copper tubing was packed with 20% silicone grease (DC 11) on chromasorb WAW and conditioned for 8 hours. This column was used for the determination of OHT in the FRH fluid.

A 21.336-decimeter (7-ft) length of 6-mm (0.25 in.) diameter copper tubing was packed with Porapak Q and conditioned for 2 hours. This column was used for the determination of H_2O.

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3 FED SPEC VV-B-680B, *Brake Fluid, Automotive,* 7/20/72.

4 ASTM D-1744, *Water in Liquid Petroleum Products by Karl Fisher Reagent.*

5 Sherma Zweig, *Handbook of Chromatography,* Vol. 1 & 2, CRC Press, 1972.

6 D. Nogare & Juvet, *Gas-Liquid Chromatography Interscience,* April 1966.

OPERATING CONDITIONS

	For Silicone Grease Column	For Porapak Q Column
Detector-thermal conductivity		
Detector cell temp, °C	295	295
Detector cell current, ma	150	150
Helium flow at exit, cc/min	80	85
Programed temperature analysis		
Isothermal temperature, °C	60	150
Terminal temperature, °C	300	–
Column heating rate, °C/min	10	–
Injection port temperature, °C	340	340

III. EXPERIMENTAL

3. **Analysis of Residual OHT.** Using the silicone grease (DC 11) column, the sample was prepared so that analytical information is obtained by relating the position and magnitude of the petroleum oil contaminant to that of the internal standard. The internal standard used was n-Decane (Practical), $CH_3\ (CH_2)_8\ CH_3$, boiling point 56-57°C.

The FRH hydraulic fluid was weighed (0.500 ± 0.05 gram) with the internal standard at 2 percent by weight of the total sample weight. A 4-$\mu\ell$ sample is injected, and the chromatograph develops at a programing rate of 10°C/minute. With an attenuation setting of X4, a 4-$\mu\ell$ sample will give excellent definition of eluted peaks. A representative chromatogram is shown in Figure 1. To quantitatively determine the residual OHT component, the following formula is used:

$$\% \text{ OHT Contaminant} = \frac{S \times W' \times F \times 100}{I \times W''}$$

where:

S = area of component to be determined
I = area of internal standard
W′ = weight of internal standard
W″ = weight of hydraulic fluid
F = correction for detector response

Note: Detector response is a function of molecular weight and chemical structure. Variable response is obtained from compounds having the same mass but different

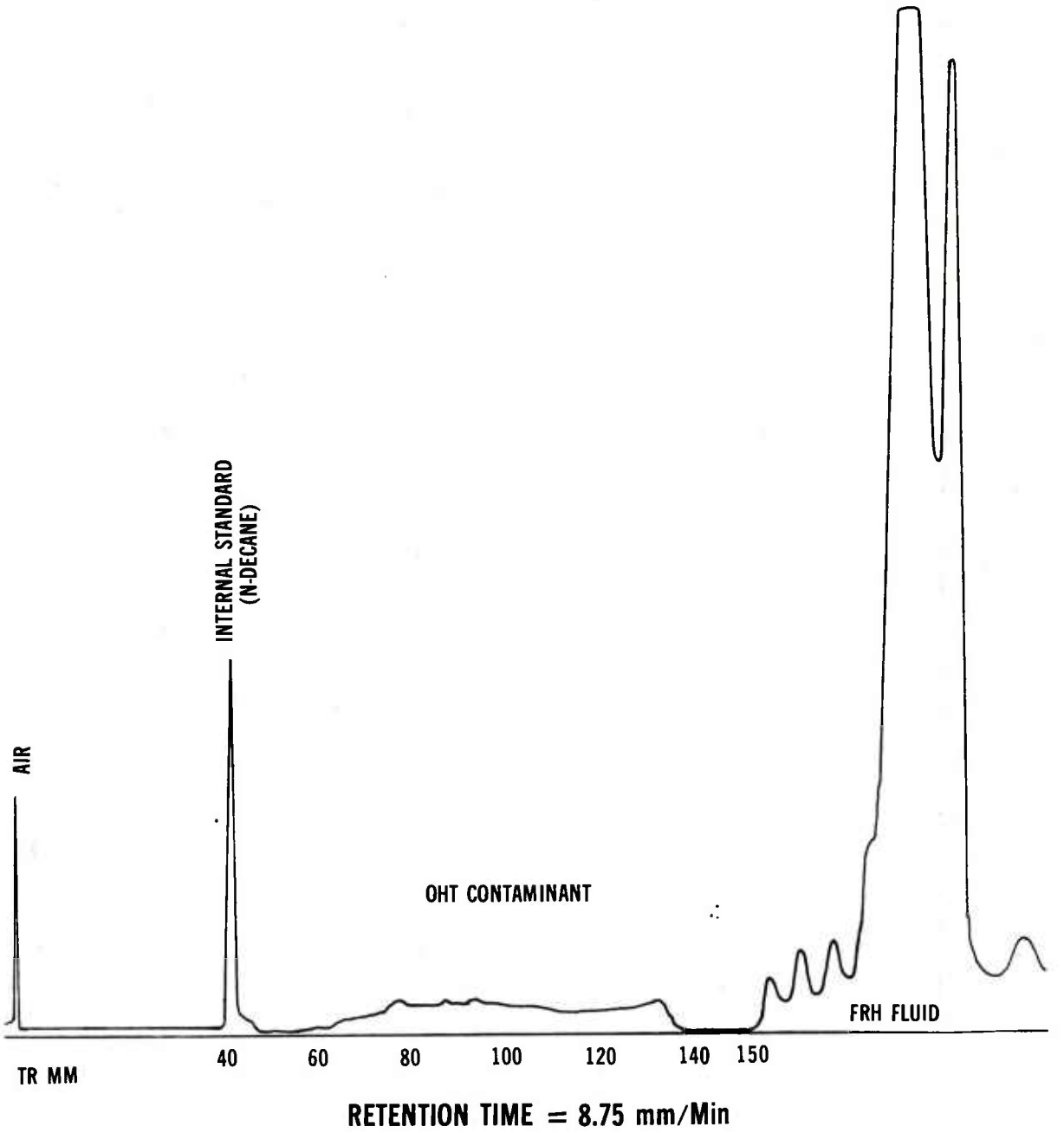

Figure 1. OHT contamination in FRH.

structures. Correction factors are established by weighing and chromatographing pure mixtures of the hydraulic fluid and internal standard; the area/mass relationship of the FRH hydraulic fluid and internal standard is put on an equivalent basis by the application of a factor to the hydraulic fluid area.

4. **Determination of Water.** The determination of water in the FRH fluid was evaluated using FRH samples taken from the fluid humidification test. A representative chromatograph is shown in Figure 2.

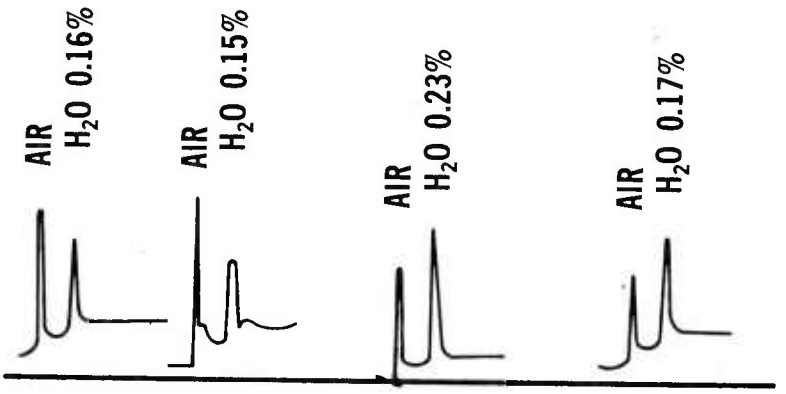

Figure 2. Water content in FRH fluid.

A 4.0 ± 0.2-$\mu\ell$ sample to be analyzed is injected into the Gas Chromatograph which is operated isothermally at 150° C. After the chromatograph develops, the peak height is measured in millimeters. Following elution of the water peak, the column temperature is increased to 250° C to elute the FRH fluid; this requires approximately 15 minutes. Column life with Porapak Q is satisfactory for approximately only 20 tests because of eventual column overload. The peak height vs percent water present using this technique is shown in Figure 3. The percent water in FRH fluid standards was prepared by addition of known amounts of water to a sample of FRH fluid obtained from a hermetically-sealed quart container which was placed in a desiccator to prevent any further water absorption. In essence, this procedure does not determine the "absolute" water content of the FRH fluid. It does permit, however, the determining of percent water increase resulting from water being adsorbed into the fluid during exposure to the humidification environment (80% relative humidity).

IV. RESULTS OF TEST

5. **Residual OHT Determinations.** Samples of used FRH fluid taken from new M60A1 (AOS) tanks operating in Germany were subsequently analyzed. The results obtained are shown in Table 1. As a result of questions relative to the repeatability of this technique, additional experiments were conducted. The degree of accuracy was

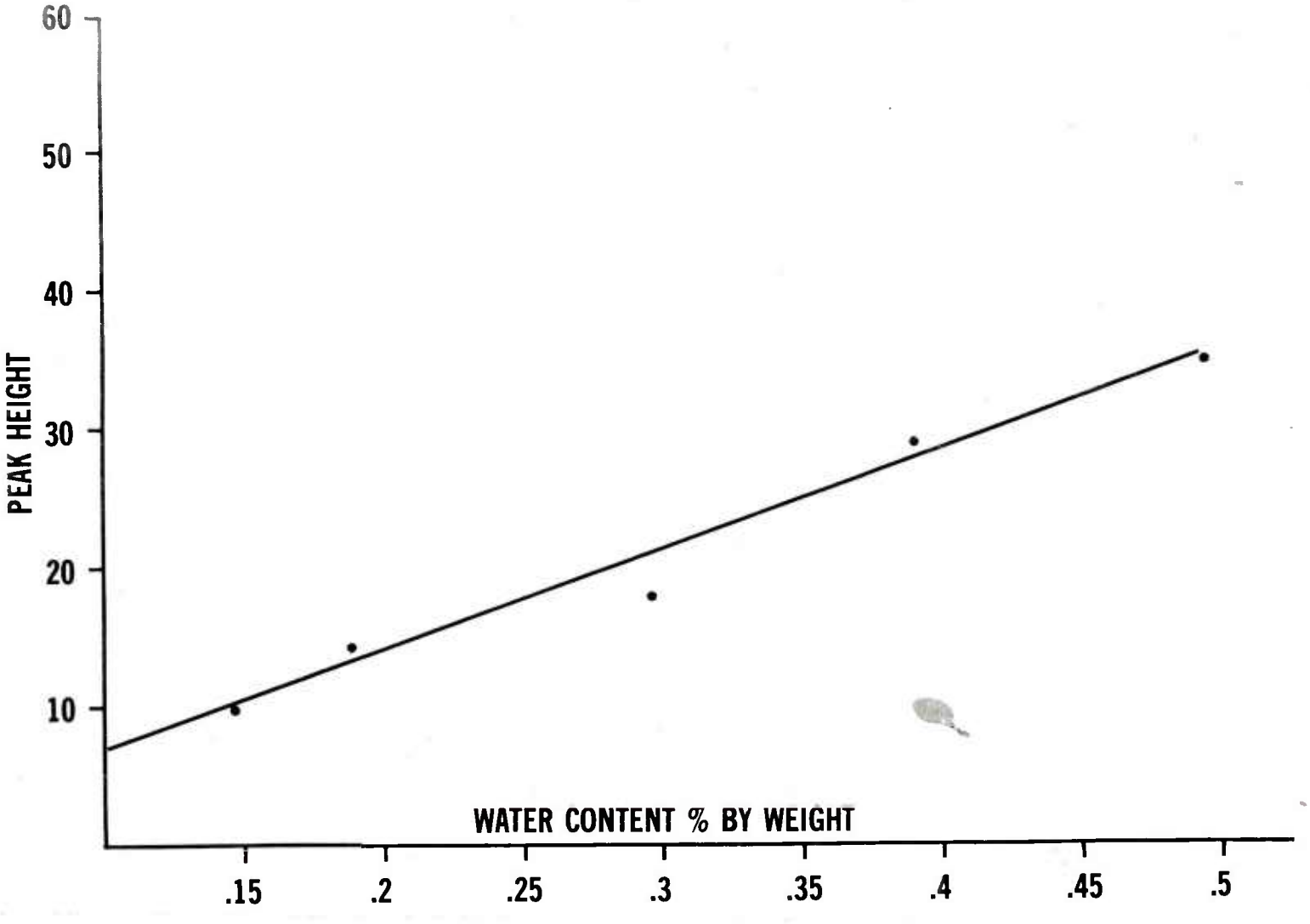

Figure 3. Water content (% wt) vs peak height (mm).

Table 1. Determination of OHT Contaminant in FRH Fluids from Fielded Tanks in Germany (%)

FRH sample from tank	Test #1	Test #2	Avg
C-16	Nil	Nil	Nil
SN6828	1.2	1.1	1.1
SN6801	0.4	0.6	0.5
SN6873	1.3	1.3	1.3
SN6822	Nil	Nil	Nil
SN6799	Nil	Nil	Nil
SN6783	1.9	1.7	1.8
SN7243	1.5	1.5	1.5
SN6853	2.0	2.6	2.3
SN6768	3.1	3.6	3.3
SN6791	4.1	4.4	4.2
SN7232	1.0	1.5	1.2
SN6862	1.3	1.9	1.6
SN6829	1.6	1.4	1.5
SN6788	1.4	1.9	1.6
SN6775	1.6	2.0	1.8
SN7225	0.5	0.5	0.5
SN6723	0.9	0.7	0.8
SN6838	1.3	1.6	1.4
SN6800	1.7	2.1	1.9

formulated from the "Square Method of Linear Equation."[7] A linear regression equation and coefficient of correlation were determined as in Figure 4. The highly linear results demonstrate the accuracy of the method over the concentration range of known samples of weight and volume percent (Table 2). Repeatability at 95% confidence level is 0.39. This was determined from five repeated tests conducted on a known sample with a concentration of 8.9%. Following this, additional samples of FRH from field tests were analyzed for residual OHT (Table 3).

[7] Roessler Alder, *Probability and Statistics*, W. H. Freeman and Co., 4th Edition, 1968.

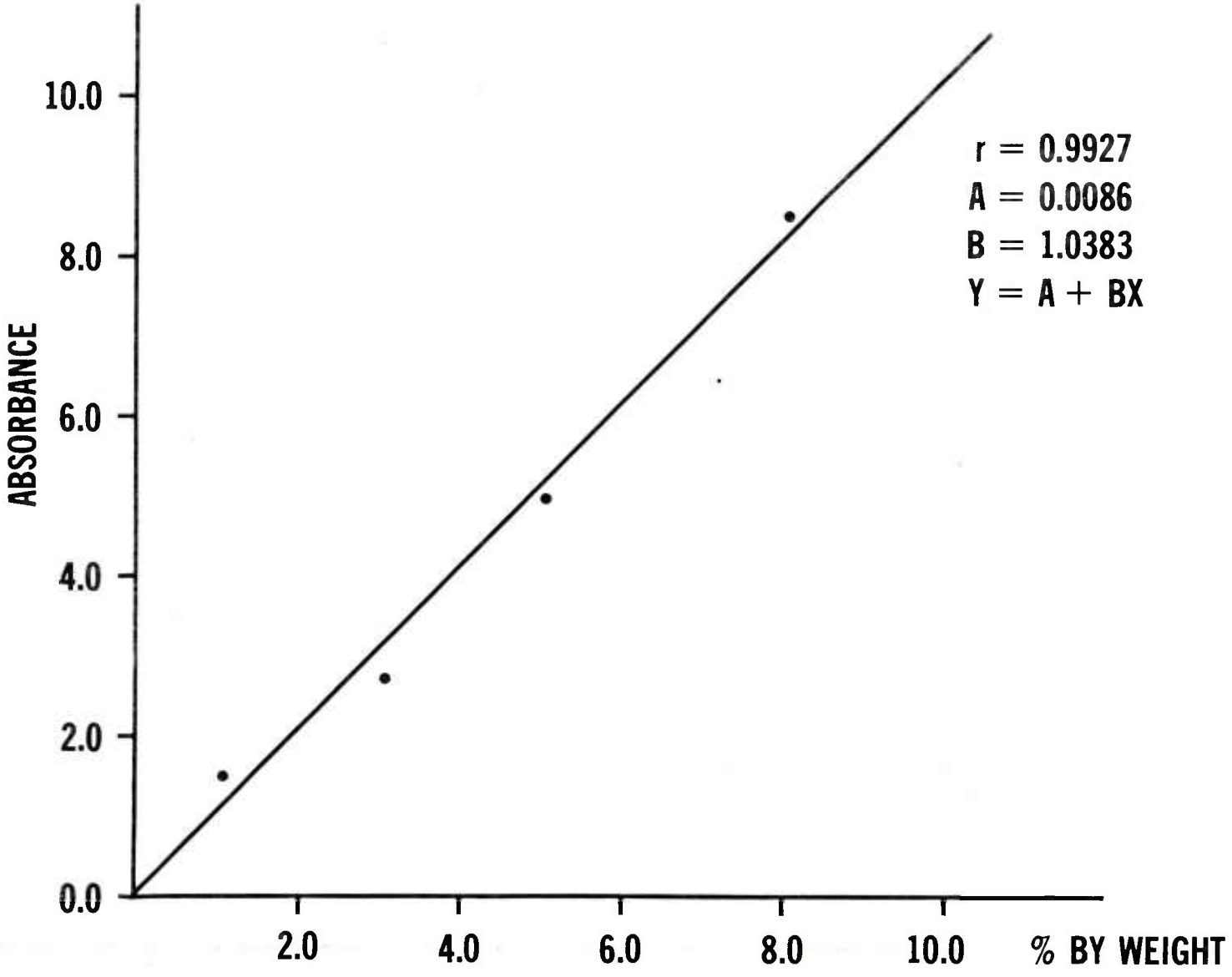

Figure 4. Square method for linear equation.

Table 2. Analysis of FRH Samples Containing Known Levels of OHT Contamination

Known (wt %)	Determined (%)	Avg Value (%)	Known (Vol %)	Determined (%)	Avg Value (%)
1	1.47		1	0.82	
1	1.40			0.92	
		1.43			.87
3.4	3.1		3	3.3	
3.4	3.0			2.8	
3.4	3.2				3.0
3.4	3.5		5	5.1	
3.4	3.1			4.8	
		3.1			5.0
5.9	5.7		8	7.4	
5.9	5.8			7.5	
5.9	5.6				7.5
5.9	5.9				
5.9	5.4				
		5.7	–	–	–
8.9	8.8				
8.9	8.8				
8.9	8.6				
8.9	8.9				
8.9	8.8				
		8.8	–	–	–

Table 3. Determination of OHT Contaminant in FRH Samples from M60A1 (AOS) Tanks Involved in Revalidation Fleet Test Program

Location	FRH Fluid Sampled From	WT % OHT Present
Ft. Bliss	Tank S/N 5923	15.0
Ft. Bliss	Tank S/N 5143	2.5
Ft. Bliss	Tank S/N 5082	13.2
Ft. Bliss	Tank S/N 5082	24.2
Ft. Bliss	Tank S/N 5923	20.2
Ft. Bliss	Tank S/N 5143	Nil
Ft. Hood	Tank S/N 5764	2.1
Ft. Hood	Tank S/N 5767	2.0
Ft. Hood	Tank S/N 2855	7.8
Ft. Hood	Tank S/N 2855	3.7
Ft. Hood	Tank S/N 5784	3.2
Ft. Hood	Tank S/N 5767	2.3
Ft. Knox	Tank S/N 5795	2.2
Ft. Knox	Tank S/N 6459	3.9
Ft. Knox	Tank S/N 2851	2.1
Ft. Knox	Tank S/N 6488	10.6
Ft. Knox	Tank S/N 6459	2.3
Ft. Knox	Tank S/N 2851	2.0
Ft. Knox	Tank S/N 5795	0.8
Ft. Knox	Tank S/N 6459	Nil
Ft. Knox	Tank S/N 2851	1.2
Ft. Knox	Tank S/N 6488 (March 76)	13.1
Ft. Knox	Tank S/N 6488 (May 76)	14.1
Ft. Polk	Tank S/N 6970	3.9
Ft. Polk	Tank S/N 6964	3.6
Ft. Polk	Tank S/N 6910	6.4
Ft. Polk	Tank S/N 6930	3.7
Ft. Polk	Tank S/N 6970	2.4
Ft. Polk	Tank S/N 6910	3.5
Ft. Polk	Tank S/N 6964	Nil

An important factor to be considered is the elution of the OHT which elutes as a "mound." The retention time for this elution is taken from the air peak at a distance from 60 mm to 145 mm. It was also found that when a standard is made with the internal standard it is necessary to run the sample immediately because of the loss of n-Decane. It was further found that the detector response factor varied with the concentration of OHT contamination. There is no interference from the synthetic base fluid or any of its additives. The repeatability results obtained are also attributed to the accuracy used in preparation and injection of the sample.

6. **Determination of Water.** The determination of water technique was primarily directed to the humidification procedure wherein the degree of water adsorption was of paramount interest. To illustrate this useful application, samples of FRH fluid were initially analyzed after 30- and 60-day intervals to assess the percent water increase due to adsorption. The results of these analyses are shown in Table 4.

Table 4. Water Increase Determinations in FRH Due to Adsorption in Humidification Test

Sample	Sample Peak Height (mm)	Sample Peak Height Before Test (mm)	% Water Increase
	After 30 Days		
FA PD-5136, Supplier A (Lot 5)	14	3.5	0.215
MIL-H-83282	2	2	Nil
FA PD-5136, Supplier B	23.5	2	0.29
FA PD-5136, Supplier A (Lot 6)	17.0	2	0.215
	After 60 Days		
FA PD-5136, Supplier A (Lot 5)	10.7	3.5	0.15
MIL-H-83282	13	2	0.16
FA PD-5136, Supplier B	23	2	0.30
FA PD-5136, Supplier A (Lot 6)	14.7	2	0.22
MIL-H-83282 + 2.5% wt (Active Ingredient) BDNS*	24.5	8	0.24

* Barium Dinonylnaphthalene Sulfonate.

The relative percent water in FRH fluid is determined, and the reproducibility is calculated by the same technique used for the OHT fluid. There is no interference from either OHT or FRH. The peak is perfectly symmetrical; and with meticulous care in measuring the height, the results are reproducible.

V. CONCLUSION

The methods developed during this investigation provided the capability for an accurate analysis of OHT contamination in FRH fluid and the relative determination of water content. This project proved that the changeover of OHT to FRH in M60A1 (AOS) turret and gun-control systems should be carefully monitored to insure against inadequate flushing procedures yielding high levels of residual OHT. With the eventual implementation of these methods, the evaluation of further field problems should be easier to resolve. These analytical techniques should be utilized to continually monitor the quality of FRH fluids during future retrofit programs.

APPENDIX

HUMIDIFICATION PROCEDURE

Two bowl-form glass desiccators, 250 mm inside diameter, having matching tubulated covers fitted with No. 8 rubber stoppers, are charged with 450 ± 25 grams of reagent grade ammonium sulfate and 125 ± 10 ml of distilled water. The surface of the salt slurry shall lie within 45 ± 7 mm of the top surface of the desiccator plate. A corrosiveness test jar* is placed in each of the desiccators, and the desiccators are placed in an area where temperature is controlled at 23 ± 1.1° C (73.4 ± 2.0° F) for 24 ± 4 hours. After the conditioning period, the rubber stoppers in the desiccator covers are carefully removed and 100 ± 1 ml of the test fluid is placed in each corrosion test jar by means of a pipette. The rubber stoppers are immediately placed back in the cover openings. The desiccators are left in the controlled temperature area for 30 or 60 days. On completion of the exposure to the humid atmosphere in the desiccators, the test jars containing the fluid are removed and tightly covered. Samples are removed by pipette, and water determinations are conducted.

* Test jar may be obtained from SAE, 400 Commonwealth Drive, Warrendale, Pennsylvania 10596.

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